



Performance and hydration characteristic of dark white evolution (DWE) cement composites blended with clay brick powder

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Abstract

Clay Bricks powder (CB) behavior on hydration characteristics of white cement pastes composites containing 5, 10, 20 and 25 mass % of CB (as substitution) was studied. Kinetic of hydration till 28 days was examined for all the blends e.g.: Whiteness Reflection (Ry), setting, Free Lime (FL) and Compressive mechanical strength (CS) were all checked up. Scanning electron microscopy (SEM) techniques have been implemented to investigate the behavior, microstructure and features of CB-pastes composites. The substitution of 5.0wt. % CB has improved the compression mechanical strength, free lime content and microstructure features of white cement pastes composite. CB has a substantial impact on the performance of white cement composite pastes, reducing whiteness reflection (Ry), and setting times while enhancing mechanical strength, bulk density and gel to space ratio. The microscopic scanning showed the existence of a thicker and more finer microstructure from calcium silicate hydrate products leads to decrease in porosity of all white composites forming extra CSH (micro scale) tightening the skeleton matrix structure responsible for the enhancement of the mechanical strength and physical properties, as well as the original morphology of calcium mono-carboaluminate hydrate. It was strongly advised that the white cement composite include 95 percent + 5 percent CB has better performance than white ordinary cement, and it adequate optimal blend, it showed a high dense compact microstructure consisting mainly of microcrystalline fibrous group with a large proportion decrease the porosity of the blend. M-CB5 represent a new type of white cement can be used as Dark White Evolution (DWE) cement for various construction purposes and reduce the cost of WC with good chance of solid waste recycling.

Keywords: Clay Bricks (CB); White Cement, Whiteness Reflection (Ry), SEM microstructure and Dark White Evolution (DWE) cement

1. Introduction

Clay bricks have a set of advantages that push many individuals and companies to adopt it in construction, including the following: heat preservation, save the energy of homes during the winter, and keep them cool during the summer; it absorbs heat from the sun's rays during the day, and transmits it at night [1-3]. Hardness and cohesion, these two features give clay bricks the ability to withstand bad weather conditions, including fires and high winds and not harming the environment: High demand for clay bricks leaves large amounts of environmental pollutants, and these huge quantities are subject to lower degradability. Ease of manufacture: the basic materials for the manufacture and processing of clay bricks are abundant, which makes the production process easy, and its low financial cost facilitates the process of repairing it. In addition, clay bricks waste are recyclable. The continuous manufacture of clay bricks can cause the fertile soil to be depleted, and its area to be greatly degraded, which negatively affects

agriculture and environment. There is a growing dependence on the building and construction materials industry on the reused /recycled supplemental cementitious materials (SCMs), solid wastes and industrial by-products for the production of eco-cements which is recently preferred in green and sustainable synthesis of construction/building materials [4-6]. Utilization of by-products generated from industrial plants such as clay bricks waste, Ground Blast Furnace Slag (GGBS), ceramic waste and many other solid wastes are possibly applicable a for minimizing the clinker/cement ratio content and the production cost subsequently. The focus toward minimizing this ratio is attributed to that the clinker is the densest component of carbon dioxide in the cement process. For example, blending pastes comprising of OPC with 10-30 wt%. GGBS or 10-20 wt% ashes commercial present in the market according to the standards of [GB-175, 2015, EN 197-1, 2011 and ASTM C-989]. Despite of, owing to accelerate the consumption rates by the building

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Receive Date: 30 December 2021, **Revise Date:** 12 January 2022, **Accept Date:** 13 January 2022

DOI: 10.21608/EJCHEM.2022.113836.5169

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materials industry and other consuming sectors, as well as restricted supply, it is projected that appropriate SCMs will be out of use in the near future [7]. Additionally, the strict adoption of climate change agreements worldwide in COP 2021, has jeopardized the sustainability of many cement plants, hence reduction in the produced amount of fly ash. Consequently, an increasing dependence on the import of SCMs and green cement have [8] been reported in several developed countries. Alternative SCMs, which are widely distributed worldwide, were not preferred. Recently, research and commercial interest has been focused on limestone-calcined clay cement (LC3) and green cement owing to that developing of a high replacement ratio up to 40.0 wt% could achieve equivalent or even better results than OPC and ready-mix when designed with a carefully approved pozzolans substitution and the availability of the main precursors (i.e., clay, limestone and sand) worldwide make LC3 as low carbon content and an alternative cementitious material. Supplementary Cement Material (SCM) is classified into two types: natural SCM (zeolite, white sand, and volcanic tuff) and synthetic SCM (clay bricks ware, silica fume and slags) [9-12]. They may significantly improve the mechanical hydraulic properties, in addition to, the characteristics and half life time of cement and concrete microstructure, due to their pozzolanic interaction [6, 13-17]. The implementation of pozzolanic materials in cement/concrete is economic due to the partial substitution of a large percentage of Portland cement with low-cost industrial by-products, resulting in lowering of the greenhouse gases generated during cement production and also they enhance the durability and operability of the final product. Moreover, increasingly blending of pozzolan with OPC creates added value by converting huge quantities of useless industrial solid wastes into sustainable building materials. The features of the developed blended cement are mostly depend on the selected filler microstructure, which are, the distribution, type, matrix, pores sizing and reactions products. The merits of adding pozzolans are the high compressive mechanical strength, great workability and durability mostly attributable to pozzolanic reactions as calcium hydroxide is totally reacted to produce extra C-A-H and C-S-H reaction products. However, the pozzolanic materials effect on the strength of the cement generally develops in later stages of hardening, depending on the pozzolanic activity. By changing the pH of the cement, the pozzolanic reaction can minimize the risk of expanding alkaline silica reactions between the cement and the aggregate. However, increasing alumina concentrations and lowering the solution

alkalinity decrease or inhibit the dissolution of the aggregate aluminosilicates [6, 14, 18-21]. The addition of clay bricks powder as SCM to cement/clinker completes the fine structure in the cement granulometric curve without increasing water consistency, plugs all capillary pores and enhances cement packing. As a result, numerous researches shown that adding white sand to cement reduces water demand [22, 23], setting times, and TP [24], while increasing combined water percent [25, 26] and compressive mechanical strength up to 17% of cement composites. Clay bricks powder enhances the rate of hydration at an early age, which causes a high early compression results, and interacts with phases of ferrite and aluminate in WC to develop monocarboaluminate [8, 27]. The inclusion of clay bricks powder can lead to shape some calcium carbosilicate hydrate and a reduction of the potential cement material (thinning effect), leading to a subsequent loss of strength [25, 28, 29]. In contrast, the inclusion of clay bricks marginally rise demand for water and overall total porosity of Portland cement composites while decreasing setting durations and bulk density [30].

The influence of clay bricks powder on the hydration of gray Portland cement was well studied worldwide. In addition, there are too little works studied the hydration characteristics of white Portland cement. In contrast, there is a lake of knowledge dealing with the role of clay bricks powder on the hydration of white Portland cement. Hence, the aim of this study is to discuss the influence of clay bricks powder on the whiteness reflection (R_y), both physico-mechanical and microstructure features of white cement composites and producing a new cement type may named as dark white evolution (DWE) cement for various construction purposes and reduce the cost of white cement with good chance of solid waste recycling.

2. Materials and Methods

2.1 Materials

White Cement (WC) and Clay Brick (CB) waste are the starting raw materials used in the current study. WC [CEM I, 52.5 N] was brought from EL-Mania white cement company (ElMinia, Egypt). Clay bricks provided from Giza area, (Giza, Egypt). Clay brick was prepared by crushing followed by milling process in ball mill for 96 hrs. Continuously to obtain very fine powder and avoiding particles coagulation, then pass through $90\mu\text{m}$ sieve mesh. Blaine i.e: specific surface area of clay brick powder after milling was $4079\text{cm}^2\text{g}^{-1}$. Figure (1) shows the visual inspection of CB vs. WC as received. SEM microstructure of clay brick powder after milling

process and proves that surface area improved and become finer is shown in figure (2). Moreover, the chemical composition of used materials are tabulated in table (1) using XRF technique.

2.2 Preparation of cement paste composites and testing methods

Overall, WC was substituted by 5, 10, 20, and 25% of CB, as shown in table (2). The water/cement ratio was varied based on the weight percentage of CB mixed cement pastes. Blends were intermixed in a rotary mixer; first, WC was added to the mixer at low speed for 1 min, then at high speed for 2 min, and last, at 400 rpm, CB was added to the homo mixer for 1.5min. The patches were molded into (2.5cm×2.5cm×2.5cm) steel molds, two layers of paste were manually pressed, and then fixed in 95±2% relative humidity (RH) at ambient room temperature. After one day, the hardened cubes were de-molded and promptly cured in tap water at 96% RH until compressive mechanical strength testing periods of 2, 7, 14 and 28 days of hydration were completed. The curing conditions were set in accordance with [31], which demonstrated the beneficial effect of high RH on the performance of CB-white cement pastes. Whiteness reflection (Ry) was measured using the "Elerpho" equipment in accordance with DIN 5033 specifications [32]. Setting times of CB mixes were determined using the Vicate equipment according to specs of ASTM C191[33]. The hydration rate of CB-cements were stopped by soaking all the cubes in an acetone/methanol mixture in a [1:1] ratio for 24hs, followed by drying at 70°C oven for 2h, and a portion of the dried sample was retained for examination using SEM technique. Compressive mechanical strength (CS) was applied to three hardened white cement cubes according to specs of ASTM C190 designations[34] employing 5 tones by (Shemizitu Machine test) at a loading rate of 25 kg/min. The free lime content (F.CaO) of hardened CB-cement pastes was measured analytically by inserting 0.5gm of

dried specimen powder in 50 ml. ethylene glycol, boiling to 70 °C for 30 min in a swirling water path, and titrated with HCL (0.1N), using equation (1) [33, 35].

$$F.CaO\% = 0.56 \times V. \text{ of HCL} \dots \dots \dots (1)$$

Figure (3) shows the schematic diagram for experimental program. The microstructures using SEM technique were generated with Gemini (Sigma 500 VP, 2020 version) at PSAS faculty, Beni-Suef University. On the other hand, the present oxides that ingested CB and WC were quantified using XRF performed with a comprehensive instrument Panalytical (ARL 9900). Whiteness reflection (Ry) obtained using Elerpho instrument (PCE-WNM 100), measuring range: 0 – 120, resolution ≈0.01, repeat accuracy <0.5 and 45/0 measuring geometry.

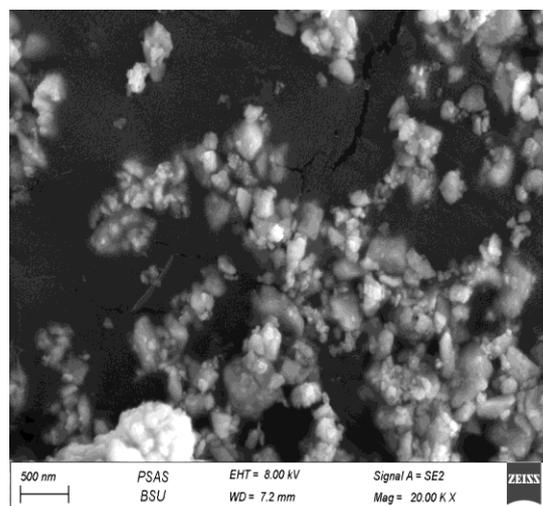


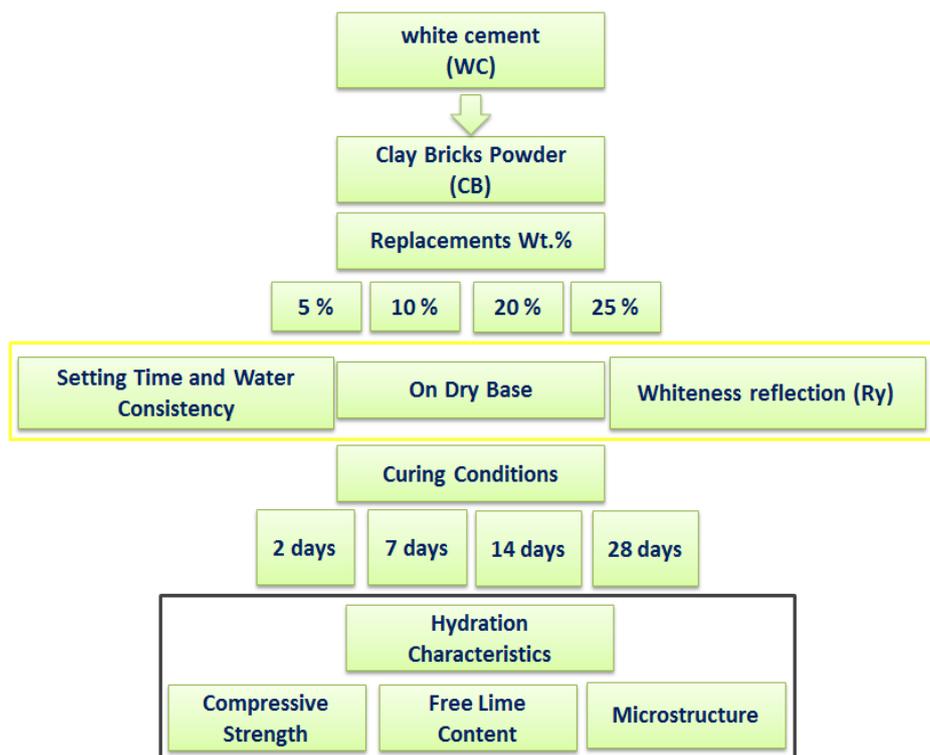
Figure (2):- SEM morphology & Microstructure of clay brick powder after milling process.

Table (1):- Chemical composition of White Cement (WC) and Clay Bricks powder (CB).

Material	SiO ₂	Al ₂ O ₃	CaO	Fe ₂ O ₃	MgO	SO ₃	Na ₂ O	K ₂ O	Cl ⁻
WC	21.76	2.85	67.77	0.11	0.23	3.05	0.41	0.31	0.07
CB	66.12	17.56	6.91	0.11	1.01	0.01	1.17	2.81	0.004

Table (2):- Mix composition of CB-white cement pastes composites.

Composites	Mix composition (Replacement)			Curing water
	WC by weight (%)	CB by Weight (%)	Total mix (%)	
M0	100.00	0.00	100.00	Tab water
M-CB5	95.00	5.00		
M-CB10	90.00	10.00		
M-CB20	80.00	20.00		
M-CB25	75.00	25.00		

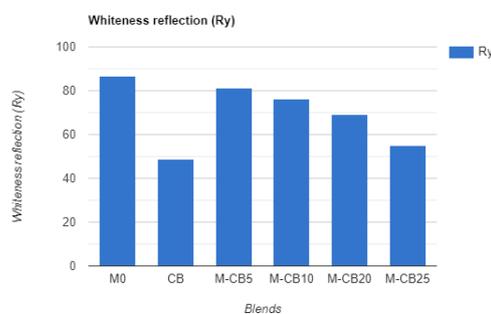
**Figure (3):-** Schematic diagram for experimental program.

3. Results and Discussions

3.1 Whiteness reflection (Ry)

CB had a very pale dark colour, so its reflection degree under the Elerpho instrument was too low, especially on the Rz axis, which administrators about the green reflection, i.e. $H_a = -1.52$ vs. $H_a = -2.03$ for WC. The whiteness reflection (Ry) with /without clay bricks powder (CB) graphically reported in figure (4) which emphasis the reduction at the mixes whiteness reflection degree (Ry) results, as clearly shown visually in figure (5) [32]. WC. It clear that, the increment of CB wt. %, leads to the (Ry) values decrement. It was followed the order: $Ry-M0 (86.80) > Ry-M-CB5 (81.10) > Ry-M-CB10 (76.08) > Ry-M-CB20 (69.37) > Ry-M-CB25 (54.96)$. The Ry degree changes from 86.80% of (M0) as WC without CB to 54.96% of (M-CB25) with the largest level of CB substitution ratio. Moreover, the (Ry) of (M-

CB5) presented the greatest value after M0 blend by 81.10%. This is owned to that the CB powder equal to 48.82% that totally impact negatively on Ry of WC blends on a dry base.

**Figure (4):-** Whiteness reflection (Ry) of white cement with or without CB (dry base).

Blends	Whiteness reflection (Ry)	Visual images
M0	$R_x = 86.94$ $R_y = 86.80$ $R_z = 81.15$ $HL = 93.17$ $Ha = -2.03$ $Hb = 4.24$	
CB	$R_x = 48.98$ $R_y = 48.82$ $R_z = 45.64$ $HL = 69.78$ $Ha = -1.52$ $Hb = 3.18$	
M-CB5	$R_x = 81.24$ $R_y = 81.10$ $R_z = 75.82$ $HL = 90.06$ $Ha = -1.90$ $Hb = 3.93$	
M-CB10	$R_x = 76.21$ $R_y = 76.08$ $R_z = 71.13$ $HL = 87.22$ $Ha = -1.81$ $Hb = 3.80$	
M-CB20	$R_x = 69.57$ $R_y = 69.37$ $R_z = 65.19$ $HL = 83.50$ $Ha = -1.78$ $Hb = 3.78$	
M-CB25	$R_x = 55.09$ $R_y = 54.96$ $R_z = 51.38$ $HL = 74.14$ $Ha = -1.61$ $Hb = 3.21$	

Figure (5):- Visual images of WC, CB and blends with whiteness reflection records

3.2 Water Consistency and Setting Times

Setting times and water consistency (W/C) of the WC composite pastes including CB are tabulated in table (3) and figure (6), illustrates the initial and final setting times of WC and CB blended white cement pastes as function of time. It was observed that the white cement composite pastes containing 5 and 10 mass % CB needs a lower water demand, 0.41 and 0.45%, respectively. Due to the active surface area of the CB powder, this cement mix has the quickest setting periods when compared to other WC-composite blends containing the CB powder [33, 36]. The released Ca^{2+} and OH^- accessible from the hydration reaction of the cement phases with the mixing tab water interact with the CB powder, resulting in an excess of calcium aluminate hydrate (C-A-H), calcium silicate hydrate (C-S-H), and calcium aluminosilicate (C-ASH) hydrated gel. The CB serves as an active crystal location for the formation of further CSH stages later on. Therefore, the setting time is reduced unlike the highest replacement blend M-CB25 which has the higher water demand i.e: 0.61%, and recorded the highest setting time results. In addition, the water consistency increment due to the high dispersion of the CB, might be explained that increasing surface area energy requires more water consistency [37, 38] because of the decrement in the fresh prepared pastes flow ability which cause an increasing in the W/C ratio and speeding up the cement hydration rate; hence, the cement pastes composites in the hybrid of 5.0 and 10.0 mass % CB possess high hydraulic characteristics. Hence, the setting time increases with increasing CB content.

Table (3):- Water Consistency and setting times of the blends

Blends	Setting time (min)		water consistency
	Initial	Final	
M0	125	145	0.37
M-CB5	140	160	0.41
M-CB10	155	180	0.45
M-CB20	210	245	0.56
M-CB25	225	290	0.61

3.3 Hydration kinetics

3.3.1 Free Lime Content

Figure (7) presents the free lime content of the CB-cement composite cured up to 28 days of hydration. Free lime content varies up and down with CB content where the free lime content decrease with increasing the curing time for replacement of CB by 5.0 and 10.0

wt% while increasing the CB above 10% resulting in increase in the free lime with increasing the curing ages this might be attributed to continuous hydration reaction which liberates calcium hydrate during the hydration period. With the addition of CB, the free lime content increases to 14 days in M-CB5 mix and subsequently decrease to 28 days in M-CB10 mix. The CB support the response rate of the cement phase hydration, which is basically the releasing rate of CH that is quicker than the consumption rate due to the pozzolanic effect with CB, increasing the FL to 7 days [39]. The CB aids in the dispersal of the WC by pushing it to a high degree of compaction and cementation and low porosity, which eases the shrinking gap between the CB and the CH freed up, resulting in the formation of additional hydration products, C-S-H, with an increase in the compressive mechanical strength. Mixtures with high content of CB (20-25%) show a high percentage of free lime due to silica and alumina which promotes the formation of C-S-H and C-A-H up to a certain extent, then act as fillers and because the Si & Al particles are large, the mixtures have high porosity and low bulk density.

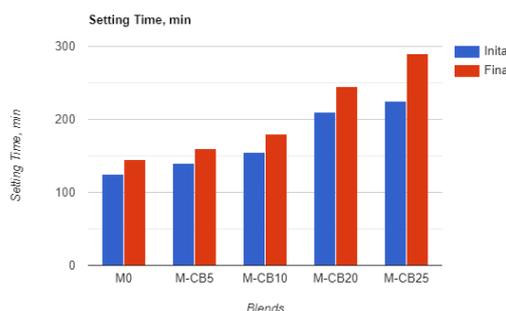


Figure (6):- Setting times of WC and CB blends

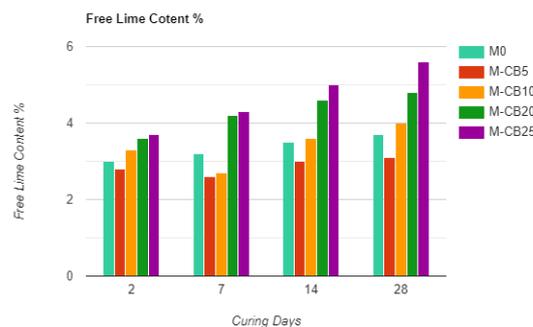


Figure (7):- Free lime content of white cement and CB blended cement pastes hydrated for 28 days.

3.3.2 Compressive strength

Effectiveness of CB replacement on the CS of the composite pastes over 28 days hydration as a function of hydration ages is represented in figure (8). The reported data showed an increasing in setting time

results in increasing in the compressive mechanical strength for all hardened white cement pastes, as the ongoing formalization of additional products of calcium aluminosilicate hydrates and calcium silicate hydrates (major source of solidification) both generated hydrates are completely deposited inside the open pores yields improvement in the competence of the specimen cement structure [40]. Adding CB (5.0%) improved the strength of the CB-composite pastes; this is due to the CB behave as a hydraulic filler which improve the matrix structure of the hardened cement blends, but also the pozzolanic effect of free Si & Al, particles, that cause the reaction of hydration with free lime and calcium hydroxide to form the gels CSH, CAH and CASH that forming the compacted and closed microstructure of the hardened white cement matrices [41, 42]. However, introducing CB is preferable to specific percentage (5.0%), higher than this value may disturb the composite microstructure and affect the matrix. Moreover, ettringite might be preserved from converting to monosulfate, thus it may leads to decrease of compressive mechanical strength effected by the internal structure stress as the abundant ettringite when the substitution of CB increased. The results also revealed that the compressive mechanical strength of the pastes of white cement composites combined with the CB is greater than that of the control sample. In contrast, the compressive strength takes the follows order: M-CB5 > M0 > M-CB10 > M-CB20 > M-CB25 when the ratio of CB blended white cement pastes decreased which is attributed to a reduction in the pozzolanic activities of the cement pastes composites. The CS of the composite cement pastes comprising of 95 % WC and 5 % CB are greater than the neat WC by 3, 7, 15, and 21 % for the specimens that cured at 2, 7, 14, and 28 days respectively.

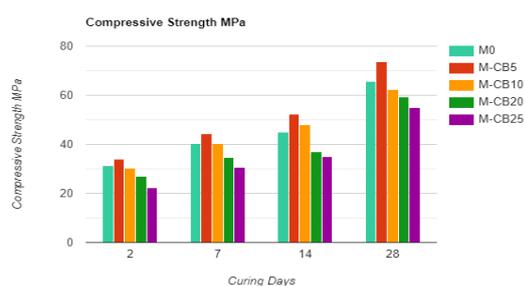


Figure (8):- Compressive Strength of white cement and CB blended cement pastes hydrated for 28 days.

3.3.3 Microstructures and phase formation

Figure (9) presents the SEM morphology and microstructure of the WC and CB cement composite cured over 28 days. The photos displays the presence of high quantity of crystalline hydrates and layers of organized hexagonal texture from CH crystals combined to clear tease and well define porous

structure. SEM depicts a excess hydrates of fiber-gel CSH responsible for crosslinking a solid and closed cement structure with increased curing life over 28 days [42, 43]. The crystalline calcium silicate hydrate shows road like particle and layers of arranged hexagonal texture of fiber-gel CH crystals are increased in M-CB5 mix as it's the optimum percent value for the CB as active pozzolanic filler material, on the other hand, these crystalline and fibrous layers are decreased with high replacement of CB as its act as complementary filler has slight hydraulic properties better than the inert fillers. The morphologically hardened specimen shows a squamous microstructure with a large number of pores filled with nanoparticles products reflect on the mechanical strength of the specimen reducing the porosity of CB pastes. Replacement up to 20 and 25 wt. % shows less dense matrix structure which might be attributed to less hydraulic properties of CB as higher replacement ratio

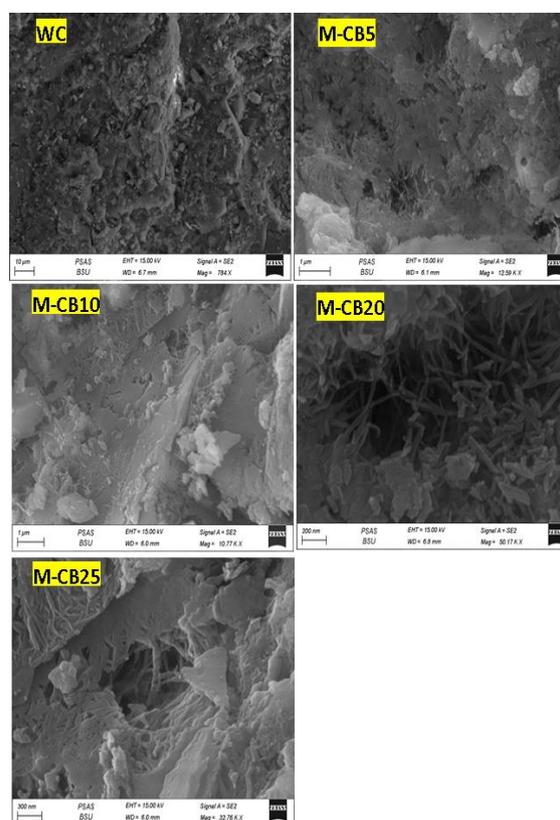


Figure (9):- Hydration and microstructure of white cement and CB blended cement pastes hydrated for 28 days

4. Recommendations

- 1) Solid waste recycling is mandatory to preserve the environment and reduce the capital cost of building and construction materials.
- 2) The use of a large part of solid waste is desirable

- to achieve green sustainability.
- 3) Dark white evolution cement opening the way for the potential application of white cement (high production cost) for use as a coating material for skin (low cost), tile manufacturing and others.
 - 4) The decrease in R_y may be difficult to detect visually so 10 wt% replacement of white cement may be viable.
 - 5) Further studies on DWE cements are required by substituting more than 40% by weight of CB powder for Masonry cement production.

5. Conclusion

DWE may represent a new type of cement for large-scale applications such as skin coating and tile manufacturing, etc...The effect of CB powder on hydration kinetics of white cement composites was evaluated. The results demonstrate that partially replacing white cement with CB filler leads to decrease in the whiteness of dry blends accordingly retarding the setting times and porosity of blends. Nevertheless, increases CS, combined water content and density of WC-CB pastes due to the pozzolanic effect that replaces clinker acting as active nuclei and increases the amount of C-S-H hydrate products. Portlandite was detected in blends hydrated for 7 days indicating the pozzolanic effect of silica and alumina at early age's of hydration whereas, C3S & C2S was detected in CB-blends hydrated up to 28 days indicating that CB effect accelerates the rate of hydration of C3S at later ages of hydration. The experimental outcomes also show that, CB powder alter the microstructure of hardened cement pastes. The morphology of C3S & C2S originally changes with progress of hydration, therefore, this study proposes a maximum content of CB as solid waste management that do not exceed 5% as a partial substitution of white cement to reach optimum cement blends performances.

6. Conflict of interest

There is no conflict of interest.

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